

ISOTROPIC CLEANSING COMPOSITION WITH BENEFIT AGENT  
PARTICLES

5      BACKGROUND OF THE INVENTION

10      1. Field of the Invention

The present invention relates to detergent compositions suitable for topical application for cleansing the human body, such as the skin and hair. In particular, it relates to isotropic compositions containing benefit agent particles.

15      2. Background of the Art

In order to be acceptable to consumers, a liquid personal cleansing product must exhibit good cleaning properties, must exhibit good lathering characteristics, must be mild to the skin (not cause drying or irritation) and preferably should even provide a benefit agent to the skin, such as moisturizers, antiwrinkle agents, skin nutrients, and the like. Several approaches have been used to provide high levels of benefit agents in a stable formula that have involved encapsulating the benefit agent which is then ruptured or dissolved with product use. For example, U.S. Patent No. 20 5,932,528 to R. Glenn, Jr., et al. issued on Aug. 3, 1999 discloses a liquid cleansing composition containing a moisturizing phase comprising an encapsulated lipophilic skin moisturizing agent and an aqueous cleansing phase comprising a surfactant and a stabilizer. The encapsulated lipophilic skin moisturizing agent comprises a lipophilic skin moisturizing agent 25 encapsulated within a complex coascervate comprising a polycation and a polyanion.

Other particles of material including microcapsules, bubbles, beads, ground particulates, and uniform particulates have been used in various cleansing and coating applications to encapsulate or bind the contents of various agents contained therein or associated therewith. For example

5 U.S. Patent No. 6,270,836 to Holman issued on August 7, 2001 describes microcapsules coated with a gel, specifically a gel produced by the sol-gel process. The gel coating provides certain resistances to the microcapsules, resulting in enhanced protection for their contents. Microcapsules containing different types of materials are known which may be used as

10 ingredients in the compositions of this invention, such as gelatin.

It is known that microcapsules may be formed by a coacervation or crosslinking process, in which lipids are coated by tiny droplets of proteins, carbohydrates, or synthetic polymers suspended in water. The process of coacervation is, however, difficult to control and depends on variables such

15 as temperature, pH, agitation of the materials, and the inherent variability introduced by a natural protein or carbohydrate.

U.S. Patent No. 6,066,613 to L. Tsaur, et al., issued on May 23,

20 2000; describes large hydrogel particles suspended in an aqueous medium and a continuous extrusion/mixing process for making this kind of hydrogel particles. The hydrogel particles comprise two different high molecular weight polymers. One is insoluble in the said aqueous medium and is used for network formation and gel integrity. The other is soluble in the said

25 aqueous medium and helps control gel swellability and gel strength. Water insoluble materials are entrapped or encapsulated inside the network formed by these two polymers and are able to be more efficiently delivered from the aqueous composition (e.g., liquid cleanser containing the hydrogel particles). However there is no disclosure or suggestion in the prior art of a

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isotropic cleansing composition containing organogel particles wherein the particles are formed by associating benefit agents that are liquids at about 75 C with a gelation agent that is a solid at about 25 C and wherein said isotropic phase composition has a viscosity of about 1,000 to about 300,000 cps at 25 C.

### BRIEF DESCRIPTION OF THE INVENTION

In one aspect of the present invention is an isotropic cleansing composition containing (a) a surfactant selected from an anionic, nonionic, amphoteric and cationic surfactant and mixtures thereof; (b) a thickening agent; (c) about 0.1 to about 25% by weight of organogel particles of from about 0.05 to about 10 millimeters in diameter, the particle comprising a benefit agent that is a liquid at about 75°C and a gelation agent that is a solid at about 25°C, the proportions of the gelation agent to benefit agent being between about 0.05% to about 70% by weight gelation agent to benefit agent, the solidification or gelation temperature of the mixture being at or above about 25°C, and (d) wherein the viscosity of the cleansing composition as measured without the organogel particles is in the range of about 1,000 to about 300,000 cps @ 1/sec shear rate at 25 C.

In another aspect of the invention is a method of depositing a benefit agent from an isotropic liquid cleansing composition, comprising the steps of:

- (a) providing said benefit agent in said cleansing composition including: (1) a surfactant selected from anionic, nonionic, amphoteric and cationic surfactants, and mixtures thereof; (2) a thickening agent, and (3) about 0.1 to about 25% by weight of

organogel particles of from about 0.05 to about 10 millimeters in diameter, the particles comprising the benefit agent that is a liquid at about 75°C and a gelation agent that is a solid at about 25°C, the proportions of the gelation agent to benefit agent being between about 0.05% to about 70% by weight gelation agent to benefit agent, the solidification or gelation temperature of the mixture being at or above about 25°C; and

(b) applying said cleansing composition to the skin or hair.

In another aspect of the invention is a method for preparing a composition containing (a) a surfactant selected from an anionic, nonionic, amphoteric and cationic surfactant and mixtures thereof; (b) a thickening agent; (c) about 0.1 to about 25% by weight of organogel particles of from about 0.05 to about 10 millimeters in diameter, the particle comprising a benefit agent that is a liquid at about 75°C and a gelation agent that is a solid at about 25°C, the proportions of the gelation agent to benefit agent being between about 0.05% to about 70% by weight gelation agent to benefit agent, the solidification or gelation temperature of the mixture being at or above about 25°C, (d) wherein the viscosity of the cleansing composition as measured without the particles is in the range of about 1,000 to about 300,000 cps @ 1/sec shear rate at 25 C., and (e) a free emollient having a weight average particle size in the range of about 1 to about 500 microns. comprising the steps of:

(a) forming a first composition having at least one surfactant selected from anionic, nonionic, amphoteric, and cationic surfactants and mixtures thereof;

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(b) adding a thickening agent to the free emollient in an amount from 1 to 50 wt. %, based on the free emollient; and

(c) mixing the base formulation of step (a) with the free emollient of step (b);

(d) adding to the first composition, the thickening agent or a blend thereof about 0.1 to about 25% by weight of organogel particles of from about 0.05 to about 10 millimeters in diameter, the particles comprising a benefit agent that is a liquid at about 75°C and a gelation agent that is a solid at about 25°C, the proportions of the gelation agent to benefit agent being between about 0.05% to about 70% by weight gelation agent to benefit agent, the solidification or gelation temperature of the mixture being at or above about 25°C.

A free emollient is herein defined as an emollient not bound to the organogel particles. Free emollient particles are also separate and distinct from organogel particles.

Organogel particles suitable for the inventive cleansing composition comprise a hydrophobic (oleophilic) phase in particulate form, with no need for a rigid shell to encapsulate the phase, and usually with no shell present. The particle's oleophilic phase contains a gelation agent, and preferably an organogelation agent. Such particles have prolonged stability and can be simply manufactured. A preferred method of manufacture of the particles comprises forming a solution of at least the oleophilic material and gelation agent at a temperature above their gelation temperature, forming droplets of the solutions, and cooling the droplets to form particulates. Cooling may be effected by exposure to ambient conditions (e.g., room temperature)

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when the ingredients are appropriately selected with regard to their melting points, or an actual cooling environment may be needed to form the particles.

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## DETAILED DESCRIPTION OF THE INVENTION

For purposes of the present invention, the following definitions are used.

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"gel" means a mixture of a solvent and solid material network (such as a solid network of particle network, fibroid network, reticulated network, and the like) wherein the solid material (e.g., any solid such as a waxy material, polymeric material, sintered or fused particle material, or any other solid material that forms a physically supportive network for the other component) is formed through physical aggregation of the solid material through any associative means. Generally, a gel is more viscous than a liquid or paste, and retains its shape when left undisturbed, i.e., is self-supporting. However, a gel is typically not as hard or firm as a wax. Gels may be penetrated more easily than a wax-like solid, where "hard" gels are relatively more resistant to penetration than "soft" gels. A rigid gel as defined herein resists deformation upon the application of a force.

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"hydrogel" means a gel in which the solvent (diluent) is water or aqueous based liquids;

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"organogel" means a gel in which the solvent (diluent) is an organic carrier or organic solvent (as opposed to water or aqueous based liquids);

"thermoreversible organogel" is synonymous with "physical organogel" and means an organogel whose network structure is due to weak, thermally unstable bonding such as hydrogen bonding (as opposed to strong, thermally stable bonds such as covalent bonds) and can,

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therefore, be heated to a free-flowing, liquid (molten) state. Upon cooling below a characteristic temperature ( $T_{gel}$ ), the bonds reform and the solid-like gel structure is re-established.

$T_{gel}$  means a temperature at which, by any physical phenomenon, a mixture of ingredients (e.g., of an oil and organic material mixed therewith) in discrete form (e.g., particulate, droplet, drop, pastille, particle, thread, fibroid, etc.) passes from a flowable or liquid condition into a stable gel or semi-solid state. Usually this occurs by solidification of gelling or organization of a liquid material into a more solid form, adding structure to the discrete form.

In one aspect of the invention is an isotropic phase cleansing composition having:

- (a) a surfactant selected from an anionic, nonionic, amphoteric and cationic surfactant and mixtures thereof;
- (b) a thickening agent; and
- (c) about 0.1 to about 25% by weight of organogel particles of from about 0.05 to about 10 millimeters in diameter, the particle comprising a benefit agent that is a liquid at about 75 C and a gelation agent that is a solid at about 25 C, the proportions of the gelation agent to benefit agent being between about 0.05% to about 70% by weight gelation agent to benefit agent, the solidification or gelation temperature of the mixture being at or above about 25 C; and
- (d) wherein the base of the inventive composition defined as the composition absent the organogel particles has a viscosity in the range of about 1,000 to about 300,000 cps @ 1/sec shear rate at 25 C as measured by a cone and plate technique described

below. Preferably the viscosity of the base is in the range of about 5,000 to 50,000 cps.

5        Preferably the inventive composition further includes a free emollient having a weight average emollient particle size in the range of about 1 to about 500 microns and about 30% by weight water. More preferably the inventive composition includes about 0.1 to about 10% by wt. of organogel particles.

10        Advantageously the gelation agent in the organogel particles includes an organic compound selected from a solid organic compound, a wax, and a polymer; and the benefit agent comprises an oil that is a liquid at about 25 C, and the like. Preferably the benefit agent is a solid at about 25 C.

15        Preferably the organogel particle has an average diameter of between about 0.1 and about 3 millimeters and the proportions of the gelation agent to benefit agent being between about 0.5% to about 50% by weight gelation agent to benefit agent; more preferably the particle has an  
20        average diameter of between about 0.1 and about 1.0 millimeters and the proportions of the gelation agent to benefit agent being between about 0.5% to about 40% by weight gelation agent to benefit agent.; and most preferably the particle has an average diameter of between about 0.1 and about 2 millimeters and the proportions of the gelation agent to benefit  
25        agent being between about 0.5% to about 30% by weight gelation agent to benefit agent. Advantageously the organogel particle is aspherical.

Preferably the gelation agent forms a network of solid gelation agent within the organogel particles formed of the benefit agent and the organogel



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particle contains a gradation of concentration of the gelation agent, with higher concentration of the gelation agent at the surface of the organogel particles than at the core of the organogel particles.

5                    Advantageously the inventive composition has less than about one percent by weight solid soap.

10                    Preferably the inventive composition includes a thickening agent added to the free emollient in amount from about 1 to about 50% wt. based on the free emollient. Advantageously the thickening agent is selected from the group consisting of polyacrylates; silica, natural and synthetic waxes; aluminum silicate; lanolin derivatives; C8 to C20 fatty alcohols; polyethylene copolymers; polyammonium carboxylates; sucrose esters; hydrophobic clays; petrolatum; hydrotalcites; cellulose derivatives, polysaccharide derivatives, mixtures thereof, and the like.

15                    Preferably the inventive composition is structured with a structurant selected from swelling clays; cross-linked polyacrylates; acrylate homopolymers and copolymers; polyvinylpyrrolidone homopolymers and copolymers; polyethylene imines; inorganic salts; sucrose esters, other  
20                    gellants; and the like. Advantageously the inventive composition contains a free emollient selected from vegetable oils, esters, animal fats, mineral oil, petrolatum, silicone oil and mixtures thereof, and the like. Preferably the free emollient is present at about 0.1 to about 15 wt % of the composition,  
25                    more preferably the free emollient functions as a carrier to deliver skin active agents (as defined below) to skin treated with the inventive composition.

Advantageously the inventive composition has about 1 to about 35 wt % of a surfactant, preferably at least about 7 wt % of the surfactant. Preferably a cosurfactant selected from betaines, amidobetaines and sulphobetaines, and the like is present.

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In another aspect of the invention is a method for preparing a composition containing (a) a surfactant selected from an anionic, nonionic, amphoteric and cationic surfactant and mixtures thereof; (b) a thickening agent; (c) about 0.1 to about 25% by weight of organogel particles of from about 0.05 to about 10 millimeters in diameter, the organogel particle comprising a benefit agent that is a liquid at about 75°C and a gelation agent that is a solid at about 25°C, the proportions of the gelation agent to benefit agent being between about 0.05% to about 70% by weight gelation agent to benefit agent, the solidification or gelation temperature of the mixture being at or above about 25°C, (d) wherein the viscosity of the cleansing composition as measured without the organogel particles is in the range of about 1,000 to about 300,000 cps @ 1/sec shear rate at 25 C., and (e) a free emollient having a weight average emollient particle size in the range of about 1 to about 500 microns, comprising the steps of:

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(a) forming a first composition having at least one surfactant selected from anionic, nonionic, amphoteric, and cationic surfactants and mixtures thereof, and the like;

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(b) adding a thickening agent to the free emollient in an amount from 1 to 50 wt. %, based on the free emollient; and

(c) mixing the first formulation of step (a) with the free emollient of step (b).

5 (d) adding to either the first composition, the thickening agent or a blend thereof about 0.1 to about 25% by weight of organogel particles of from about 0.05 to about 10 millimeters in diameter, the organogel particles comprising a benefit agent that is a liquid at about 75 C and a gelation agent that is a solid at about 25 C, the proportions of the gelation agent to benefit agent being between about 0.05% to about 70% by weight gelation agent to benefit agent, the solidification or gelation temperature of the mixture being at or above about 25 C.

10 Preferably the first composition has less than about 1 percent by weight of solid soap

15 In another aspect of the invention is a method of depositing a benefit agent from an isotropic liquid cleansing composition to the skin or hair, comprising the steps of:

20 (a) providing said benefit agent in said cleansing composition including:

1. a surfactant selected from anionic, nonionic, amphoteric and cationic surfactants, and mixtures thereof;

25 2. a free emollient having a weight average emollient particle size in the range about 1 to about 500 microns;

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3. a thickening agent, wherein the thickening agent is added to the free emollient in amount from about 1 to about 50% wt., based on the free emollient; and

4. about 0.1 to about 25% by weight of organogel particles of from about 0.05 to about 10 millimeters in diameter, the organogel particles comprising a benefit agent that is a liquid at about 75 C and a gelation agent that is a solid at about 25 C, the proportions of the gelation agent to benefit agent being between about 0.05% to about 70% by weight gelation agent to benefit agent, the solidification or gelation temperature of the mixture being at or above about 25 C, and

(b) applying said cleansing composition to the skin or hair.

Preferably the composition contains less than about 1 percent by weight of solid soap.

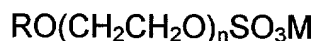
#### Surfactants:

Surfactants are an essential component of the inventive cleansing composition. They are compounds that have hydrophobic and hydrophilic portions that act to reduce the surface tension of the aqueous solutions they are dissolved in. Useful surfactants can include anionic, nonionic, amphoteric, and cationic surfactants, and blends thereof.

#### Anionic Surfactants:

The cleansing composition of the present invention contains one or more anionic detergents. The anionic detergent active which may be used may be aliphatic sulfonates, such as a primary alkane (e.g., C<sub>8</sub>-C<sub>22</sub>) sulfonate, primary alkane (e.g., C<sub>8</sub>-C<sub>22</sub>) disulfonate, C<sub>8</sub>-C<sub>22</sub> alkene sulfonate, C<sub>8</sub>-C<sub>22</sub> hydroxyalkane sulfonate or alkyl glyceryl ether sulfonate (AGS); or aromatic sulfonates such as alkyl benzene sulfonate.

The anionic may also be an alkyl sulfate (e.g., C<sub>12</sub>-C<sub>18</sub> alkyl sulfate) or alkyl ether sulfate (including alkyl glyceryl ether sulfates). Among the alkyl ether sulfates are those having the formula:



wherein R is an alkyl or alkenyl having 8 to 18 carbons, preferably 12 to 18 carbons, n has an average value of greater than 1.0, preferably greater than 3; and M is a

solubilizing cation such as sodium, potassium, ammonium or substituted ammonium. Ammonium and sodium lauryl ether sulfates are preferred.

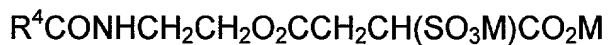
The anionic may also be alkyl sulfosuccinates (including mono- and dialkyl, e.g., C<sub>6</sub>-C<sub>22</sub> sulfosuccinates); alkyl and acyl taurates, alkyl and acyl sarcosinates, sulfoacetates, C<sub>8</sub>-C<sub>22</sub> alkyl phosphates and phosphates, alkyl phosphate esters and alkoxyl alkyl phosphate esters, acyl lactates, C<sub>8</sub>-C<sub>22</sub> monoalkyl succinates and maleates, sulphoacetates, alkyl glucosides and acyl isethionates, and the like.

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Sulfosuccinates may be monoalkyl sulfosuccinates having the formula:

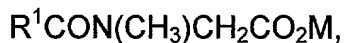


amide-MEA sulfosuccinates of the formula;



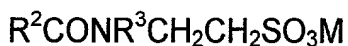
wherein  $R^4$  ranges from  $C_8$ - $C_{22}$  alkyl and M is a solubilizing cation.

Sarcosinates are generally indicated by the formula:



wherein  $R^1$  ranges from  $C_8$ - $C_{20}$  alkyl and M is a solubilizing cation.

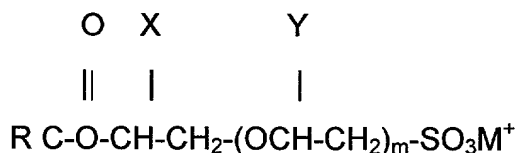
Taurates are generally identified by formula:



wherein  $R^2$  ranges from  $C_8$ - $C_{20}$  alkyl,  $R^3$  ranges from  $C_1$ - $C_4$  alkyl and M is a solubilizing cation.

Another anionic surfactant that may be used is a C<sub>8</sub>-C<sub>18</sub> acyl isethionates. These esters are prepared by reaction between alkali metal isethionate with mixed aliphatic fatty acids having from 6 to 18 carbon atoms and an iodine value of less than 20. At least 75% of the mixed fatty acids have from 12 to 18 carbon atoms and up to 25% have from 6 to 10 carbon atoms.

The acyl isethionate may be an alkoxyated isethionate such as is described in Ilardi et al., U.S. Patent No. 5,393,466, titled "Fatty Acid Esters of Polyalkoxyated isethonic acid; issued February 28, 1995; hereby incorporated by reference. This compound has the general formula:



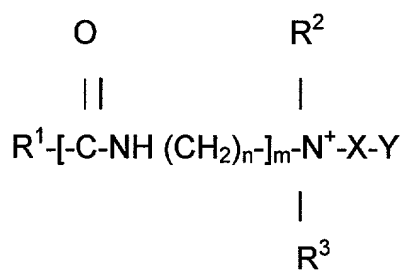
wherein R is an alkyl group having 8 to 18 carbons, m is an integer from 1 to 4, X and Y are hydrogen or an alkyl group having 1 to 4 carbons and M<sup>+</sup> is a monovalent cation such as, for example, sodium, potassium or ammonium.

#### Amphoteric Surfactants

One or more amphoteric surfactants may be used in this invention. Such surfactants include at least one acid group. This may be a carboxylic or a sulphonic acid group. They include quaternary nitrogen and therefore are quaternary amido acids. They should generally include an alkyl

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or alkenyl group of 7 to 18 carbon atoms. They will usually comply with an overall structural formula:



where  $\text{R}^1$  is alkyl or alkenyl of 7 to 18 carbon atoms;

$\text{R}^2$  and  $\text{R}^3$  are each independently alkyl, hydroxyalkyl or carboxyalkyl of 1 to 3 carbon atoms;

$n$  is 2 to 4;

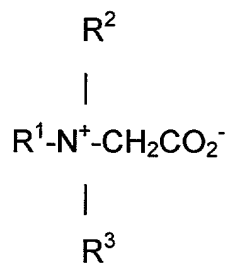
$m$  is 0 to 1;

$\text{X}$  is alkylene of 1 to 3 carbon atoms optionally substituted with hydroxyl, and

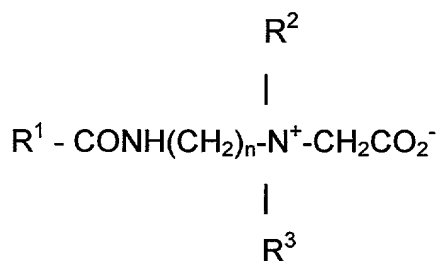
$\text{Y}$  is  $-\text{CO}_2-$  or  $-\text{SO}_3-$



Suitable amphoteric surfactants within the above general formula include simple betaines of formula:



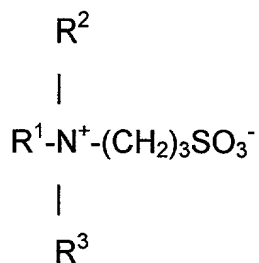
and amido betaines of formula:



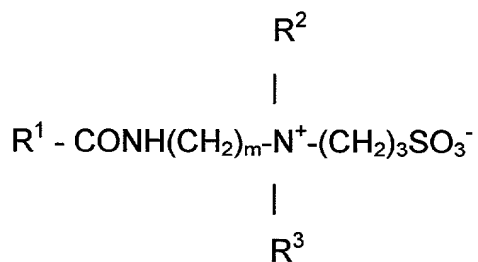
where n is 2 or 3.

In both formulae  $R^1$ ,  $R^2$  and  $R^3$  are as defined previously.  $R^1$  may in particular be a mixture of  $C_{12}$  and  $C_{14}$  alkyl groups derived from coconut oil so that at least half, preferably at least three quarters of the groups  $R^1$  have 10 to 14 carbon atoms.  $R^2$  and  $R^3$  are preferably methyl.

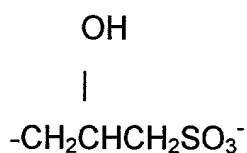
A further possibility is that the amphoteric detergent is a sulphobetaine of formula:



or



where m is 2 or 3, or variants of these in which  $-(CH_2)_3SO_3^-$  is replaced by



In these formulae  $R^1$ ,  $R^2$  and  $R^3$  are as discussed previously.

Amphoacetates and diamphoacetates are also intended to be covered in possible zwitterionic and/or amphoteric compounds which may be used such as e.g., sodium lauroamphoacetate, sodium cocoamphoacetate, and blends thereof, and the like.

### Nonionic Surfactants

5 One or more nonionic surfactants may also be used in the cleansing composition of the present invention.

10 The nonionics which may be used include in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example aliphatic alcohols, acids, amides or alkylphenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Specific nonionic detergent compounds are alkyl ( $C_6$ - $C_{22}$ ) phenols ethylene oxide condensates, the condensation products of aliphatic ( $C_8$ - $C_{18}$ ) primary or secondary linear or branched alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. Other so-called nonionic detergent compounds include long chain tertiary amine oxides, long chain tertiary phosphine oxides and dialkyl sulphoxide, and the like.

20 The nonionic may also be a sugar amide, such as a polysaccharide amide. Specifically, the surfactant may be one of the lactobionamides described in U.S. Patent No. 5,389,279 to Au et al. titled "Compositions Comprising Nonionic Glycolipid Surfactants issued February 14, 1995; which is hereby incorporated by reference or it may be one of the sugar amides described in Patent No. 5,009,814 to Kelkenberg, titled "Use of N-Poly Hydroxyalkyl Fatty Acid Amides as Thickening Agents for Liquid Aqueous Surfactant Systems" issued April 23, 1991; hereby incorporated into the subject application by reference.

Cationic Skin Conditioning Agents

An optional component in compositions according to the invention is a cationic skin feel agent or polymer, such as for example cationic celluloses. Cationic cellulose is available from Amerchol Corp. (Edison, NJ, USA) in their Polymer JR (trade mark) and LR (trade mark) series of polymers, as salts of hydroxyethyl cellulose reacted with trimethyl ammonium substituted epoxide, referred to in the industry (CTFA) as Polyquaternium 10. Another type of cationic cellulose includes the polymeric quaternary ammonium salts of hydroxyethyl cellulose reacted with lauryl dimethyl ammonium-substituted epoxide, referred to in the industry (CTFA) as Polyquaternium 24. These materials are available from Amerchol Corp. (Edison, NJ, USA) under the tradename Polymer LM-200.

A particularly suitable type of cationic polysaccharide polymer that can be used is a cationic guar gum derivative, such as guar hydroxypropyltrimonium chloride (Commercially available from Rhone-Poulenc in their JAGUAR trademark series). Examples are JAGUAR C13S, which has a low degree of substitution of the cationic groups and high viscosity, JAGUAR C15, having a moderate degree of substitution and a low viscosity, JAGUAR C17 (high degree of substitution, high viscosity), JAGUAR C16, which is a hydroxypropylated cationic guar derivative containing a low level of substituent groups as well as cationic quaternary ammonium groups, and JAGUAR 162 which is a high transparency, medium viscosity guar having a low degree of substitution.

Particularly preferred cationic polymers are JAGUAR C13S, JAGUAR C15, JAGUAR C17 and JAGUAR C16 and JAGUAR C162, especially Jaguar C13S. Other cationic skin feel agents known in the art

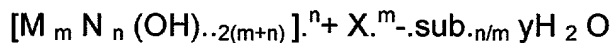
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may be used provided that they are compatible with the inventive formulation.

### Thickening Agents

Suitable thickening agents can be added either directly to the free emollient or as a structurant for the composition or both. Suitable thickening agents for the free emollient include polacrylates; fumed silica natural and synthetic waxes, alkyl silicone waxes such as behenyl silicone wax; aluminium silicate; lanolin derivatives such as lanesterol; C8 to C20 fatty alcohols; polyethylenecopolymers; polyammonium stearate; sucrose esters; hydrophobic clays; petrolatum; hydrotalcites; and mixtures thereof, and the like.

Hydrotalcites are materials of general formula:



where

M is a divalent metal ion e.g.  $Mg^{2+}$  ;

N is a trivalent metal ion e.g.  $Al^{3+}$  ;

X is an exchangeable anion e.g.  $CO_3^{2-}$ ,  $NO_3^-$ , stearate, cinnimate;

m is the number of divalent metal ions; and

n is the number of trivalent metal ions.

Particularly preferred thickening agents for the free emollient include silica, alkyl silicone waxes, paraffin wax C8 to C20 fatty alcohols, petroleum jelly and polyethylenecopolymers, and the like.

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While some materials can function as both a free emollient and a thickener therefor it will be appreciated that the emollient and thickening function cannot be provided by the same component. However, it will be understood that where the composition comprises two or more free emollients one of said free emollients could also function as a thickening agent.

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Preferably the amount of thickening agent is from about 4 to about 25% by weight based on the level of free emollient.

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Although the compositions of the invention may be self- structuring preferably they will also comprise a structurant, i.e. a material added to increase the viscosity at zero shear. Suitable materials include swelling clays, for example laponite; fatty acids and derivatives hereof and, in particular fatty acid monoglyceride polyglycol ethers; cross-linked polyacrylates such as Carbopol (.TM.) (polymers available from Goodrich); acrylates and copolymers thereof, polyvinylpyrrolidone and copolymers thereof; polyethylene imines; salts such as sodium chloride and ammonium sulphate; sucrose esters; gellants; and mixtures thereof, and the like.

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Of the clays particularly preferred are synthetic hectorite (laponite) clay used in conjunction with an electrolyte salt capable of causing the clay to thicken. Suitable electrolytes include alkali and alkaline earth salts such as halides, ammonium salts and sulphates, and the like.

As mentioned above compositions according to the invention may also comprise a thickening agent in addition to the thickening agent added to the free emollient, i.e. a material which maintains the viscosity of the composition as the shear rate thereof is increased during use. Suitable materials include cross-linked polyacrylates such as Carbopol (.TM.) (polymers available from Goodrich); fatty acids and derivatives thereof, and the like, and, in particular, fatty acid monoglyceride polyglycol ethers; natural gums including alginates, guar, xanthan and polysaccharide derivatives including carboxy methyl cellulose and hydroxypropyl guar; propylene glycols and propylene glycol oleates; salts, and the like, such as sodium chloride and ammonium sulphate; glycerol tallowates; and mixtures thereof, and the like.

Further examples of structurants and thickeners are given in the International Cosmetic Ingredient Dictionary, Fifth Edition, 1993, published by CTFA (The Cosmetic, Toiletry & Fragrance Association), incorporated herein by reference.

#### Cationic Surfactants

One or more cationic surfactants may also be used in the cleansing composition.

Examples of cationic detergents are the quaternary ammonium compounds such as alkyldimethylammonium halogenides.

Other suitable surfactants which may be used are described in U.S. Patent No. 3,723,325 to Parran Jr. titled "Detergent Compositions

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Containing Particle Deposition Enhancing Agents" issued March, 27, 1973; and "Surface Active Agents and Detergents" (Vol. I & II) by Schwartz, Perry & Berch, both of which are also incorporated into the subject application by reference.

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In addition, the inventive cleansing composition of the invention may include 0 to 15% by wt. optional ingredients as follows: perfumes; sequestering agents, such as tetrasodium ethylenediaminetetraacetate (EDTA), EHDP or mixtures in an amount of 0.01 to 1%, preferably 0.01 to 0.05%; and coloring agents, opacifiers and pearlizers such as zinc stearate, magnesium stearate,  $\text{TiO}_2$ , EGMS (ethylene glycol monostearate) or Lytron 621 (Styrene/Acrylate copolymer) and the like; all of which are useful in enhancing the appearance or cosmetic properties of the product.

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The compositions may further comprise antimicrobials such as 2-hydroxy-4,2', 4' trichlorodiphenylether (DP300); preservatives such as dimethyldimethylhydantoin (Glydant XL1000), parabens, sorbic acid etc., and the like.

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The compositions may also comprise coconut acyl mono- or diethanol amides as suds boosters, and strongly ionizing salts such as sodium chloride and sodium sulfate may also be used to advantage.

25

Antioxidants such as, for example, butylated hydroxytoluene (BHT) and the like may be used advantageously in amounts of about 0.01% or higher if appropriate. Antioxidants may also be present in concentrations effective to be skin active agents.



Humectants such as polyhydric alcohols, e.g. glycerine and propylene glycol, and the like; and polyols such as the polyethylene glycols listed below and the like may be used.

5	Polyox WSR-205	PEG 14M,
	Polyox WSR-N-60K	PEG 45M, or
	Polyox WSR-N-750	PEG 7M.

10           The free emollient "composition" may be a single free emollient component or it may be a mixture of two or more compounds one or all of which may have a beneficial aspect. In addition, the free emollient itself may act as a carrier for other components one may wish to add to the cleansing composition.

15           A blend of a hydrophobic and hydrophilic free emollients may be used. Preferably, hydrophobic emollients are used in excess of hydrophilic emollients in the inventive cleansing composition. Most preferably one or more hydrophobic emollients are used alone. Hydrophobic emollients are  
20           preferably present in a concentration greater than about 10 % by weight, more preferably about 12 % by weight. The term "emollient" is defined as a substance which softens or improves the elasticity, appearance, and youthfulness of the skin (stratum corneum) by either increasing its water content, adding, or replacing lipids and other skin nutrients; or both, and  
25           keeps it soft by retarding the decrease of its water content.

Useful free emollients and benefit agents include the following:

- (a) silicone oils and modifications thereof such as linear and cyclic polydimethylsiloxanes; amino, alkyl, alkylaryl, and aryl silicone oils;
- (b) fats and oils including natural fats and oils such as jojoba, soybean, sunflower, rice bran, avocado, almond, olive, sesame, persic, castor, coconut, mink oils; cacao fat; beef tallow, lard; hardened oils obtained by hydrogenating the aforementioned oils; and synthetic mono, di and triglycerides such as myristic acid glyceride and 2-ethylhexanoic acid glyceride;
- (c) waxes such as carnauba, spermaceti, beeswax, lanolin, and derivatives thereof;
- (d) hydrophobic and hydrophilic plant extracts;
- (e) hydrocarbons such as liquid paraffins, vaseline, microcrystalline wax, ceresin, squalene, pristan and mineral oil;
- (f) higher fatty acids such as lauric, myristic, palmitic, stearic, behenic, oleic, linoleic, linolenic, lanolic, isostearic, arachidonic and poly unsaturated fatty acids (PUFA);
- (g) higher alcohols such as lauryl, cetyl, stearyl, oleyl, behenyl, cholesterol and 2-hexydecanol alcohol;
- (h) esters such as cetyl octanoate, myristyl lactate, cetyl lactate, isopropyl myristate, myristyl myristate, isopropyl palmitate, isopropyl adipate, butyl stearate, decyl oleate, cholesterol isostearate, glycerol monostearate, glycerol distearate, glycerol tristearate, alkyl lactate, alkyl citrate and alkyl tartrate;
- (i) essential oils and extracts thereof such as mentha, jasmine, camphor, white cedar, bitter orange peel, yru, turpentine, cinnamon, bergamot, citrus unshiu, calamus, pine, lavender, bay, clove, hiba, eucalyptus, lemon, starflower, thyme, peppermint, rose, sage, sesame, ginger, basil, juniper, lemon grass, rosemary, rosewood, avocado, grape, grapeseed, myrrh, cucumber, watercress, calendula, elder flower, geranium,

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linden blossom, amaranth, seaweed, ginko, ginseng, carrot, guarana, tea tree, jojoba, comfrey, oatmeal, cocoa, neroli, vanilla, green tea, penny royal, aloe vera, menthol, cineole, eugenol, citral, citronelle, borneol, linalool, geraniol, evening primrose, camphor, thymol, spirantol, penene, limonene and terpenoid oils;

(j) lipids such as cholesterol, ceramides, sucrose esters and pseudo-ceramides as described in European Patent Specification No. 556,957;

(k) vitamins, minerals, and skin nutrients such as milk, vitamins A, E, and K; vitamin alkyl esters, including vitamin C alkyl esters; magnesium, calcium, copper, zinc and other metallic components;

(l) sunscreens such as octyl methoxyl cinnamate (Parsol MCX) and butyl methoxy benzoylmethane (Parsol 1789);

(m) phospholipids;

(n) antiaging compounds such as alpha hydroxy acids, beta hydroxy acids; and

(o) mixtures of any of the foregoing components, and the like.

Preferred free emollients and benefit agents are selected from triglyceride oils, mineral oils, petrolatum, and mixtures thereof. Further preferred emollients and benefit agents are triglycerides such as sunflower seed oil.

#### Isotropic Micellar Phase Compositions:

The inventive cleansing composition possesses isotropic micellar phase microstructure. The rheological behavior of all surfactant solutions, including liquid cleansing solutions, is strongly dependent on the

microstructure, i.e., the shape and concentration of micelles or other self-assembled structures in solution.

When there is sufficient surfactant to form micelles (concentrations above the critical micelle concentration or CMC), for example, spherical, cylindrical (rod-like or discoidal), spherocylindrical, or ellipsoidal micelles may form. As surfactant concentration increases, ordered liquid crystalline phases such as lamellar phase, hexagonal phase, cubic phase or L3 sponge phase may form. The non-isotropic hexagonal phase, consists of long cylindrical micelles arranged in a hexagonal lattice. In general, the microstructure of most personal care products consist of either an isotropic dispersion including spherical micelles; and rod micelles; or an ordered liquid crystalline phase such as a lamellar dispersion.

As noted above, micelles may be spherical or rod-like. Formulations having spherical micelles tend to have a low viscosity and exhibit Newtonian shear behavior (i.e., viscosity stays constant as a function of shear rate; thus, if easy pouring of product is desired, the solution is less viscous. In these systems, the viscosity increases linearly with surfactant concentration.

Rod micellar solutions are more viscous because movement of the longer micelles is restricted. At a critical shear rate, the micelles align and the solution becomes shear thinning. Addition of salts increases the size of the rod micelles thereof increasing zero shear viscosity (i.e., viscosity when sitting in bottle) which helps suspend particles but also increases critical shear rate (point at which product becomes shear thinning; higher critical shear rates means that the product is more difficult to pour).

Lamellar dispersions differ from both spherical and rod-like micelles because they can have high zero shear viscosity (because of the close packed arrangement of constituent lamellar droplets), yet these solutions are very shear thinning (readily dispense on pouring). That is, the solutions can become thinner than rod micellar solutions at moderate shear rates.

In formulating liquid cleansing compositions, therefore, there is the choice of using isotropic micellar phases such as rod-micellar solutions; or lamellar dispersions. When rod-micellar solutions are used, they also often require the use of external structurants to enhance viscosity and to suspend particles. For this, carbomers and clays are often used. At higher shear rates (as in product dispensing, application of product to body, or rubbing with hands), since the rod-micellar solutions are less shear thinning, the viscosity of the solution stays high and the product can be stringy and thick.

One way of characterizing isotropic micellar dispersions (hereinafter "isotropic compositions") include cone and plate viscosity measurement as described below. The inventive isotropic composition absent the organogel particles has a viscosity in the range of about 1,000 to about 300,000 cps @ 1/sec shear rate at 25 C as measured by a cone and plate technique described below. Preferably the viscosity is in the range of about 5,000 to 50,000 cps.

### ORGANOGELES

A stable particle suitable for the inventive composition of from 0.05 to 2, 5 or 10 millimeters in diameter comprises a mixture of a material that is

an oleophilic liquid at 25°C, 50°C, or 75°C and a gelation agent or organogelation agent, the proportions of the gelation agent to oleophilic liquid being between 0.05% to 70%, or 0.05% to 50%, or 0.05% to 30% by weight gelation agent to oleophilic liquid, the  $T_{gel}$  of the mixture being above 25°C. The gelation agent may comprise an organogelation agent, which is a term understood in the art and further defined herein. The particle may provide the oleophilic material liquid (or solid) as an oil, oil mixture, oil dispersion or suspension, or oil solution. A group of particles may have an average diameter of between 0.05 to 10 millimeters, between 0.08 and 10 millimeters, between 0.08 and 5 millimeters, between 0.1 and 5 millimeters, between 0.1 and 2 millimeters, and 0.1 and 1 millimeters and the proportions of the gelation agent to oleophilic liquid may be, for example, between 0.5% to 20% or more by weight gelation agent to oleophilic liquid.

The  $T_{gel}$  of the mixture or the  $T_{gel}$  of the individual components forming the organogel particle refers to the temperature at which the composition gels from an essentially liquid state as the temperature drops, or the temperature at which an individual component gels when the temperature of a liquid component is dropped.

A preferred method of forming such a stable organogel particle may include:

- a) mixing together at least the oleophilic liquid and the gelation agent,
- b) providing the mixture as a fluid material (having liquid or flowable properties), the mixture being at a temperature at least 5°C above the solidifying temperature, such as at least 5°C above the  $T_{gel}$  of the mixture and/or the gelation agent;
- c) forming discrete elements, such as droplets, pastilles, strands, particles, shapes, or other solid accumulations or gels of the material (preferably a fluid material); and

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d) cooling the solid accumulation (e.g., droplets, strands, particles, shapes, etc.) to a temperature at or at least 5°C below the solidification temperature of the gelation agent of the  $T_{gel}$  of the mixture or gelation agent or at any other temperature that causes gelation to form solid, relatively stable particles.

Organogel particle hardness is an important parameter in formulating an acceptable cleansing composition. Typically the hardness is advantageously adjusted to be satisfactory to the user and can be defined as the Particle Breakage Index (PBI) or the amount of force (weight) required to break or rupture the particle. PBI is conveniently expressed in terms of grams per square millimeter. and is calculated based on the following equation:

$$PBI = \text{weight need to break a particle (g)} / \text{cross section area of particle (mm}^2\text{.)}$$

Preferably, the particles should have a PBI value of about 0.05 up to 10, more preferably about 0.5 to about 5 and most preferably about 1.5 to 3.0. Such properties correspond to what the average user of the inventive cleansing composition considers as highly acceptable hardness for the particles. The strength can be controlled by the exercise of reasonable judgment and selection of ingredients and proportions according to art recognized techniques. Viscosity agents, thixotropic agents, surfactants, solid binders, antistatic agents, crosslinking agents, coupling agents, dispersing agents, emulsifying agents, thinning agents, and the like are among the types of additives that can be added in amounts between 0.001 and 30% by weight of the hydrophobic (oleophilic) material (e.g., the oil) to

assist in the control of the properties of the organogel particles, such as size, physical strength, durability, and the like.

With respect to particle gel rheology, typically, gels possess a storage modulus  $G'(w)$  which exhibits a pronounced plateau at higher frequencies (on the order of 1-100 radians/second), and a loss modulus  $G''(w)$  which is considerably smaller than the storage modulus in the plateau region. In a strict sense, the term "gel" applies to systems having a value  $G'(w)$  that is higher than its value of  $G''(w)$  at low frequencies. Many of the compositions according to the present invention are gels by one or both of the above definitions. A gel is free-standing or self-supporting in that its yield value is greater than the shear stress imposed by gravity.

Rheological parameters such as the storage modulus  $G'(w)$  can be measured as a function of angular frequency with a parallel-plate rheometer. For example, such parameters can be generated using a Rheometrics Dynamic Analyzer Model 70, using a 0.5 cm stainless steel plate and a 2.3 mm sample gap, over a temperature sweep of 25-85°C at 1% strain and 6.3 radians/sec. Characterization of the rheological behavior of a gelled body according to the present invention can be made using the Rheometrics instrument and conditions set forth above. As the gel is heated, it retains significant gel-like character until its  $T_{gel}$  temperature is reached. The viscosity of the particles at room temperature may typically vary from 10 to a few thousand cP or higher, even to the point of approaching properties that appear more solid than gel like.

Another property that may be directly measured is shear viscosity at e.g. 0.1 rad/sec as measured by ASTM D 2765-Procedure A. The shear viscosity, for example, may range from at least about 5 percent higher compared with the oil at room temperature to orders of magnitude higher



than the shear viscosity at room temperature of the oils as measured by ASTM D 2765-Procedure A.

5 The precise process and phenomena that contribute to the formation of the particles, and the precise structure of the particles may vary between different combinations of materials, and even between different proportions of the same materials. It is believed that at least one of the following three systems and processes occur during the practice of the present invention, although these descriptions and/or hypothesis are not intended to be limiting on the scope of actual processes and structures that are provided in the practice of the present invention. The first method is where the two organic materials (the oleophilic agent and the gelation agent) are provided as a mixture at a temperature at which both materials are together as a fluid (e.g., preferably as a true liquid, but at least in a form that is flowable and in which the at least two ingredients are intimately mixed). This temperature usually requires that the softening temperature and/or the flow temperature or melt temperature (e.g.,  $T_{g1}$  and/or  $T_{g2}$ ) be exceeded. After the fluid and the intermixed components have been provided at this elevated temperature (e.g., at a temperature above 25°C, above 40°C, above 50°C, above 60°C, above 75°C, above 85°C, above 100°C, above 120°C, above 150°C, above 175°C, and the like, up to temperatures short of where the individual components boil or decompose), the composition is cooled. The cooling temperature, depending upon how low a temperature to which the mixture is dropped, will cause at least one or both of the at least two ingredients to solidify. This cooling is done while the mixture is in or being placed into particulate form, as by prilling, pastilling, spray drying (or spray cooling in this case), or any other particle forming process. Because the at least two materials are intermixed, as opposed to the encapsulating relationship found in prior art microencapsulation processes,

as the first or both at least two materials (the oleophilic agent and the gelation agent) harden or solidify, they remain intermixed. It is particularly at this point that the nature of the particle forming phenomenon becomes alternative in nature. The gelation agent, in some circumstances is believed to form fibroids, tendrils, fibers, reticulated structures, aspherical shapes, elongate elements or the like (e.g., with aspect ratios of at least 3, aspect ratios of at least 4 or 5, and higher, up to continuous filamentary elements with extremely high aspect ratios) within the particle material (e.g., within a droplet). These various structural elements form a physical support for the oleophilic material, whether supporting the oleophilic material by surface tension to the solid, adsorption to the solid, partial entrapment by the solid, entrapment by the solid, partial absorption by the solid, or by any other physical or physical chemical means by which the solid material supports and maintains a particulate shape for the at least two materials. A second method of stable particle, or droplet formation may occur where the structural elements may even be particulates without elongate structure (e.g., aspect ratios between 1 and 3, or between 1 and 5), wherein the physical forces between the particles and the oleophilic agent sustains the particle, or droplet structure. The particles may or may not be in contact with each other and may or may not be bound by physical or chemical means to each other during the gelation or solidifying process, but they do provide actual support for the particle, or droplet structure so that the product exists as a stable particle. A third method by which particle stabilization or gelation may occur is with the formation of a gradation of materials within the particle, with the highest concentration of the gelation agent at the surface of the droplet and the least concentration of the gelation agent (yet still above 0.0) occurring at center of the particle. This is not traditional encapsulation or microencapsulation where there is a clear distinction and sharp separation between a shell (solid) and a core (liquid).

The gradation may have the outermost surface of the particle as 100% gelation material (and entrapped solids or intended diluents or active ingredients), but may also have 99%, 95%, 90%, 80%, 75%, 50%, 40%, 35% 30% or the like of gelation agent in the outermost surface and lower amounts within the body of the particle, the remainder of the concentration being provided as the oleophilic material and its associated components.

As noted above and later herein, a preferred aspect of the invention uses a gelation agent that is a solid at 25°C to stabilize an oleophilic fluid, flowable or liquid material. The oleophilic material may also be a solid or difficult to flow material at 25°C, so that the particle may even be a solid oleophilic material in a solid matrix or network of solid supporting elements or gelation agent. The solid network of gelation agent may have provided structural support for the oleophilic material during the solidification process and remains as a network after the cooling of the oleophilic material to a solid. The final stable particle may therefore be a support system of the gelation agent and a liquid or semisolid or solid oleophilic material; in particle form. It must be noted again, that the term particle generically refers to a bead, pastille, solid droplet, fibroid, sphere, oblong, filamentary object, reticulated network, or other solid form with the required maximum diameter of 10 mm or less. The average diameter may be based upon number average diameter or any other basis as desired.

According to the present invention, molten (liquid) organogels are formed into any solid accumulation of materials such as droplets, particles, pastilles, strands, fibroids, from liquids provided above their gelation temperatures ( $T_{gel}$ ). As is understood in the art, the  $T_{gel}$  is the temperature at which gel-to-sol transition occurs. It is preferred that the  $T_{gel}$  of the molten compositions be about between 20°C and 70°C when used to form the

solid accumulations in forming the particles or the invention. It is also preferred that the molten coating compositions be coated from about 5C<sup>0</sup> to 25C<sup>0</sup> above the T<sub>gel</sub> of the composition.

5           The particle-forming compositions of the invention may also form thermoreversible gels, although any type of gel formation may be used. Generally, a thermoreversible organogel is characterized by the observation of a T<sub>gel</sub>. The T<sub>gel</sub> may be determined by several different criteria, such as, for example, the temperature at which: (a) when a liquid  
10           composition is cooled, there is a rapid, discrete, qualitative change from liquid to solid properties; (b) when a liquid composition is cooled, there is a sudden increase in hydrodynamic radius, as measured by dynamic light scattering methods; (c) when a liquid composition is warmed, a 1 mm drop of mercury will flow through the composition; and (d) the elastic and viscous  
15           moduli are equivalent.

          As noted above, the particles (which term is used to generically encompass the organogel particles of the invention, whether they are beads, strands, particles, shapes, fibroids, filamentary shapes, pastilles or  
20           any other solid accumulation) are formed from two distinct materials that may be present as two distinct phases. The two distinct materials comprise what are referred to in the practice of the invention as the organic solid network, and the other component is referred to as the oleophilic liquid, oleophilic material or generally as the oleophilic organic medium. It may be  
25           difficult at times to distinguish which material is apparently working at the various function because of the dimensions and intimacy of the materials. For example, two phase or multiphase systems may form, or what is generally referred to as interpenetrating networks, where the functions of the materials, particularly where both materials may be present as solids,

cannot clearly be defined or distinguished as a supporting, carrier, or supported phase. Even where a solid organic network is supporting a liquid organic medium, chilling the particle so that the liquid organic medium solidifies does not remove the particle from the practice of the invention.

5 The gelation agent or organic solid network may be any organic material that passes from liquid to solid state during the cooling process and with its solid structure supports the other organic medium. The organic solid medium may comprise organic materials that are solids at the cool-down temperature, organic or inorganic waxes, polymers, copolymers, oligomers, and the like. The term gelation is used, rather than some other term, because the process appears to act in the manner of a gel-forming process, and many of the particles appear to act as gels, rather than solid particles.

10 The nature of some of the compositions, such as those that can be defined according to standards in the art as thermoreversible gels, are clearly gel and gelation compositions. Non-limiting examples of liquid compositions that form thermoreversible organogels at or near room temperature include nitrocellulose in ethyl alcohol, and the like. Although not wishing to be bound by theory, Applicants postulate that

15 thermoreversible organogels suitable for use in the present invention may contain a polymer or copolymer wherein the polymer or copolymer chain contains two or more different functional groups or discrete regions, e.g., syndiotactic sequences prone to crystallite formation in a solvent or solvent mixture. It is believed that the addition of an alcohol to a polymer capable of

20 hydrogen bonding prevents or reverses gel formation because of the hydrogen bonding of alcohol-based solvents with polymer sites capable of hydrogen bonding. The requirements of the solvent blend are that it must not interact with polymer hydrogen bonding sites along the polymer chain and thereby interfere with the polymeric binder's ability to undergo

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hydrogen bonding with itself through the such sites, yet it must solvate the polymer at the non-hydrogen bonding sites and be an overall solvent for the polymer at temperatures above  $T_{gel}$ . A further requirement is that upon cooling below  $T_{gel}$  the polymer remains in solution forming a gel that is a homogeneous, clear, solid solution as opposed to forming an opaque heterogeneous mass.

In using molten thermoreversible organogel solutions, it is necessary to form the particles at temperatures above the  $T_{gel}$  of the organogel. On the other hand, it is desirable to perform the processing at the lowest possible temperature above  $T_{gel}$  in order to facilitate rapid onset of gelation after forming. It has been found advantageous to provide a "chill-box" or similar rapid chilling mechanism which functions immediately after the forming operation to trigger rapid gelation to inhibit interlayer mixing. Preferably, the molten organogel temperatures during forming should be  $5C^{\circ}$  to  $25C^{\circ}$  above  $T_{gel}$ . More preferably, the molten organogel temperatures during coating should be from about  $10C^{\circ}$  to about  $15C^{\circ}$  above  $T_{gel}$ .

The coating solutions or dispersions are solidified organogels at or near room temperature and liquids at a modestly elevated temperature. The solutions are warmed to  $5C^{\circ}$  to  $25C^{\circ}$  above their  $T_{gel}$  so that they are liquids. The molten solutions are spray dried, prilled, pastilled, dispersion solidified, drip-dried, spray extruded, or as known by those of ordinary skill in the art to be otherwise formed into a solid particle by being cooled below their gel temperature while in particulate or droplet form.

The term "PLURONIC<sup>TM</sup>" refers to poloxamer compounds useful as gelation agents and which are sold collectively under the trademark

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PLURONIC™ (BASF, Parsippany, N.J.). PLURONIC F-127 (PL 127) corresponds to poloxamer 407, a polyoxypropylene-polyoxyethylene block copolymer described by Schmolka in the Journal of Biomedical Materials Research 6:571-582, 1972. Other PLURONIC™ compounds may be used in the present invention. As used in this application, the terms PLURONIC™ organogel, poloxamer organogel, and polyoxyethylene/polyoxypropylene organogel are synonymous.

Other non-limiting examples of gelation materials include waxes (e.g., beeswax, paraffin, water-insoluble wax, carbon-based wax, silicone wax, microcrystalline wax, etc.), triglycerides, acid triglycerides, polymers, fluoroalkyl (meth)acrylate polymers and copolymers, acrylate polymers, ethylene/acrylate copolymers, polyethylene, polypropylene polymers and copolymers, fatty acids, fatty alcohols, fatty acid esters, fatty acid ethers, fatty acid amides, alkylene polyhydric alcohols, fatty acid amide of an alkanolamine, glyceryl monostearate, (aryl-substituted)sugars, dibenzyl sorbitol (or mannitol, rabbitol, etc.), condensates and precondensates of lower monohydric alcohols, trihydroic alcohols, lower polyglycols, propylene/ethylene polycondensates, and the like.

The oleophilic material may be any oleophilic material, whether a single pure compound, a solution, a composition, a mixture, an emulsion (e.g., an oil-in-water emulsion or preferably a water-in-oil emulsion), a dispersion, or the like. Among the many types of oleophilic materials useful in the inventive composition may be included oils, emollients (as described above), fragrances, cosmetics, colorants ; and skin active materials such as antimicrobial agents, medications, exfoliating agents, astringents, antioxidants, enzymes, sunscreens or ultraviolet radiation absorbing compositions, and the like. As noted the general range, when considering

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two phases, ranges from about 0.5% to 70% by weight of gelation agent and from 70 to 99.5% by weight liquid oleophilic composition. Alternative ranges of the gelation agent are from 1-60%, 1-50%, 1-30%, 2-25%, 5-25%, 7-25%, 10-25%, and 12-20% by total weight of the particle or by weight of the hydrophobic liquid. At the higher levels of gelation agent (e.g., from 12%-30%, greater than 15%, and from 15-30% by weight), the particles tend to be gelled more firmly, and are very stable,

Benefit agents may be pure, raw, may contain dispersed materials or may have suspended or dispersed materials added to the oil. For example, abrasive particles may be suspended to provide scrubbing or exfoliant effects, silica, titania, calcium carbonate, talc, starch, pigments, conductive particles, reflective particles, frangible particles, reactive materials, moisture sensitive particles (e.g., urea particles, zeolites, gas-generating particles), and the like. Other forms of active or useful ingredients are described elsewhere. The concentration of such materials may be from 0%, 0.001% to 20% or 40% of the hydrophobic component.

Various other ingredients, some of which have been noted above, may also be included within the solution prior to being formed into droplets. Such additional ingredients include (a) a wax or wax mixture of about 1 part by weight mineral ester wax having an acid value of about 0 to about 55, about 4 parts by weight partly saponified mineral ester wax having an acid value of about 10 to about 45, about 1.5 parts by weight insect wax having an acid value of about 0.2 to about 24; (b) a film-forming agent that is a curable material selected from the group consisting of a curable emulsion polymer, a curable resin, a curable aminofunctional silicone, and mixtures thereof; (c) a film-modifying agent that is a surfactant selected from the group consisting of a surface-active aminofunctional silicone, a linear



arylalkyl modified polydialkyl siloxane, a linear alkylated copolymer of vinylpyrrolidone with a long chain (C<sub>12</sub> to C<sub>22</sub>) alpha olefin, and mixtures thereof; (d) a nonionic emulsifying agent having an HLB\* value of about 10 to about 15 and, for example, selected from the group consisting of an oil-soluble polyglycerol ester of a hydrophobic fatty acid capable of forming a water in oil emulsion, a water-soluble C<sub>8</sub> to C<sub>18</sub> alkylphenol ether with ethylene oxide having an average number of ethylene oxide units of of about 5 to about 70, and mixtures thereof (\*HLB-hydrophile-lipophile balance); (e) an effective amount of an anionic oleophilic dispersing agent; (f) a thickening agent selected from the group consisting of polymers, colloids, alkaline earth metal aluminum silicate, non-ionic, cationic or anionic esters, and mixtures thereof; (g) an organic solvent selected from the group consisting of a liquid aliphatic hydrocarbon, a liquid aromatic hydrocarbon, and an oleoresinous liquid having an average kauri-butanol value above 50, and mixtures thereof; and (h) an effective amount of a preservative.

The properties of the particles may be controlled by various disciplines and additives as desired. The size can be controlled by the size of spray heads, the degree of shearing forces, the temperatures of the initial gel solution, the gelation rate, the viscosity of the gel composition, and other physical mechanisms.

The invention will now be described in greater detail by way of the following non-limiting examples. The examples are for illustrative purposes only and not intended to limit the invention in any way.

Except in the examples, or where otherwise explicitly indicated, all numbers in this description indicating amounts or ratios of materials or

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conditions or reaction, physical properties of materials and/or use are to be understood as modified by the word "about".

Where used in the specification, the term "comprising" is intended to include the presence of stated features, integers, steps, components, but not to preclude the presence or addition of one or more features, integers, steps, components or groups thereof.

All percentages in the specification and examples are intended to be by weight unless stated otherwise.

#### Example 1.

The following components were used to form an organogel particle suitable for the inventive cleansing composition.

Sunflower oil	89.5%
Polywax® 500	10.0%
Pigment	0.5% (Sun Chemical D&C Red #6 BA Lake)

This formulation was combined and melted in a flask. The melted composition was then subsequently prilled into a water catch tank using a single fluid nozzle. Three different sized nozzles were used to produce particles that were approximately 600 to 800 microns, 800 to 1200 microns, and 1000 to 1400 microns

The above particles of the size 800 to 1200 microns were placed in a gel consisting of: water, Vitamin E in SD alcohol 40, glycerin, polysorbate 20, aloe barbadensis gel, carbomer, triethenolamine, methyl paraben, imidazolidinyl urea, fragrance, Vitamin A palmitate, tea tree oil, colorants.

#### Example 2.

The following components were used to form an organogel particle suitable for use with the inventive cleansing composition.

Sunflower oil 80.9%

Fragrance oil 9.3

Intercontinental Fragrances Cinnamon Concentrate FG#8673

Polywax® 500 9.8

These ingredients were melted together and made into pastilles by depositing pasty droplets of materials onto a release surface and drying the droplets.

#### Example 3.

The following components were used to form an organogel particle suitable for use with the inventive cleansing composition. The firmness of sunflower oil gelled with 10% Polywax® 500 was compared with the firmness of sunflower oil gelled with 10% beeswax. It was found that the sunflower oil gelled with Polywax® 500 was firmer than sunflower oil gelled with beeswax.

#### Example 4

The following kinds of organogel particles (a) to (f) suitable for use with the inventive cleansing composition were made:

(a)

Sunflower oil 70%

Beeswax 30%

(b).

Sunflower oil 70%

Carnauba wax 30%

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(c)

Polywax® 2000	10%
Sunflower oil	90%

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(d)

Koster Kuenen Synthetic Paraffin Wax #201	5%
Sunflower oil	95%

(e)

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Polywax® 500	10%
Mineral Oil	90%

(f)

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90% AC Humko Cottonseed Flakes F05030	
10% Polywax® 1000	

Blends (a) to (f) were separately melted and formed into pastilles.

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#### Example 5

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The degree of softness and spreadability as perceived with use of various inventive organogel particles was assessed according to test methods described below and the results are summarized in table 1.

Table 1.

Particle Composition	Particle Attributes	
Sunflower to Polywax Ratio	Degree of Softness	Ease of Spread
80:20	Very Hard	Unacceptable
85:15	Hard	Unacceptable
90:10	Just Right	Highly Acceptable
94:6	Soft	Slightly Acceptable

#### Example 6.

Isotropic cleansing compositions I to V were formulated as listed in table 2 and were evaluated for organogel particle stability and particle hardness. Composition I was prepared by dispersing the polyacrylate in excess water. Thereafter sorbic acid and trisodium citrate dihydrate were added to the resulting polymer dispersion. The 3 surface-active agents were mixed and the resulting mix added to the polymer dispersion. Thereafter, minor components were added. The viscosity of the resulting composition was measured at the shear rate of 1/sec at 25 C and thickener added until the required viscosity was obtained. Then the organogel particles were added to the final mix and the resultant mix filled into containers.

Compositions II & III were processed in identical manner, except for the absence of Dimethicone in Composition III. Both compositions were prepared by combining the bulk of the water with SLES and Sodium Cocoyl isethionate, the mixture heated to about 75 C and mixed until a clear

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solution was produced. Then Carbopol dispersed in water was added to the solution and subsequently neutralized with sodium hydroxide. Xanthan gum dispersion was then added to the mix. At this point, dimethicone was added (Composition II). Then the other surfactant and other minor components were added to the mix and blended together while at the same time allowing the mix temperature to cool. Perfume was added when the batch temperature cooled down to about 35C. The viscosity of the resulting composition was measured and adjusted until the desired viscosity was attained. Finally, the organogel particles were added.

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Table 2.

<u>Chemical Name</u>	<u>% Active in Formula</u>			
	I	II	III	IV
Ammonium Laureth Sulfate/Ammonium Lauryl Sulfate/Cocamide MEA/PEG-5 Cocamide Blend (25:25:5:2)	-	-	-	9.0
Cocamidopropyl Betaine	1.0	8.0	8.0	0.8
Sodium Cocoyl Isethionate	-	5.0	5.0	-
Sodium Laureth Sulfate	4.0	2.0	2.0	-
Alkylpolyglucoside	5.0			
Glycerin	-	-	-	0.5
Dimethicone (60,000 cst)	-	5.0	-	-
Thickener	~1.0	-	-	-
Carbopol Aqua SF 1 @ 30%	-	-	-	1.5
Carbopol ETD 2020	0.8	0.8	0.8	
Polyquaternium-10	-	-	-	0.1
Xanthan Gum	-	0.4	0.4	-
Organogel Particles	2.0	2.0	2.0	1.0
Opacifier/Colorant/Perfume /Diluent/Water	To 100.0	To 100.0	To 100.0	To 100.0

The organogel particles contained about 98% sunflower seed oil and Polywax 500 blend at an 80:20 ratio and the remaining 2% balance consisted of pigment colorants. The average particle size was about 1200 microns.

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Assessment of product stability was done by visual assessment based on 2-phase separation of the base and particles creaming to the top.

Particle Stability was also measured by Stoke equation (see below):

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Using this equation, the following numbers were determined for Composition I:

$$\sigma = 1010 \text{ kg/m}^3$$

$$\rho = 917 \text{ kg/m}^3$$

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$$\eta_o = 2 \times 10^6, \text{ kg/m-sec}$$

$$V_s = 3.65 \times 10^{-11} \text{ m/sec or } \sim 1 \text{ mm/year.}$$

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The 'creaming' rate of the particle at 25° C was determined to be about 1 mm/year. This clearly shows the relative stability of the base in suspending the organogel particles. Results of the product stability evaluation revealed no signs of product separation nor accumulation of particles on the product's surface. The finished products were visually stable, i.e., particles did not exhibit any signs of rising to the top and remained uniformly distributed throughout the base. The 'creaming' rate of the particle at 25° C may also be evaluated using an accelerated aging test as summarized below:

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1. 51.7 C for a period of 1 month
2. 40.5 C for a period of 1 month
3. -9.5C/25 C – 3 complete cycles where 1 cycle constitute 23.5 hours under -9.5 C followed by another 23.5 hours under 25 C
4. 40.5 C/25 C – 3 complete cycles

Stability evaluation is performed visually by comparing the samples stored under accelerated conditions and the control sample (stored under 25 C)

#### Example 7.

Organogel particles of the composition given in table 3 were made according to the following procedure: All three samples were spray chilled (prilled) using a pressure pot and single fluid nozzle. The orifice diameter used in samples A and B was 0.020 inches and the orifice diameter of the nozzle used in sample C was 0.028 inches. The material was melted and the pot was heated to keep the material above 105 degrees C. The pressure used to prill sample A and B was 20 psi and the pressure used to prill C was 40 psi.

Table 3

Sample	Composition	Sample Size, n (number of particles tested)	Mean Diameter, (microns)	Mean Weight to Break Particle (g)	Mean PBI (Particle Breakage Index, g/mm2)
A	Sunflower Oil 89.96% Polywax 500 9.99% Pigment 0.05%	15	742.90 $\mu$ $\pm 40.26$	3.2g $\pm 0.68$	1.9
B	Sunflower Oil	15	773.30 $\mu$	12.9g $\pm 2.6$	6.9

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	79.92% Polywax 500 19.98% Pigment * 0.10%		±64.74		
C	Sunflower Oil 93.54% Polywax 500 5.96% Pigment * 0.50%	31	1184.2 μ ±193.2	0.77g ±0.51	0.2

Sun Chemical Light Rubine Lake

Methods:

### Particle Breakage Index (PBI)

A microscope, a microscope slide and a microscope coverslip are required. A coverslip is placed on top of the slide. A single particle is obtained from the sample and placed on top of the coverslip. Using the microscope and a 10x objective, the diameter of the particle is measured. After measuring the particle, another coverslip is placed on top of the particle. Sufficient weight is placed on top of the coverslip until the particle ruptures. The weight required to rupture the particle is recorded. Cross sectional area of the particle is estimated as  $A = \pi r^2$ , where r is the radius of the particle.

### Particle Stability

Particle Stability may be shown when the rate of particles rising/floating ('creaming') to the top is very low. This rate is measured by Stoke equation:

$$V_s = \frac{2r^2g(\sigma - \rho)}{9\eta_o}$$

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where  $r$  = particle radius, m

$g$  = acceleration due to gravity,  $9.807 \text{ m/sec}^2$

$\sigma$  = density of suspending medium,  $\text{kg/m}^3$

$\rho$  = density of particle,  $\text{kg/m}^3$

$\eta_o$  = zero shear viscosity,  $\text{kg/m-sec}$

$V_s$  = 'creaming' rate,  $\text{m/sec}$

Perceived organogel particle softness/hardness and ease of spreadability during use.

Panelists pour organogel (about 10 to 20) particles between the thumb and either the index and/or middle finger and press them gently between the fingers until the particles rupture. The panelist then rate the particles' degree of softness/hardness based on the following rating scale – Very Hard, Hard, Just Right, Soft and Very Soft. Then the panelists squeeze the ruptured particles in circular motion between fingers.

To assess the particles' ease of spreadability, a small amount of sample (about 10 to 20 particles) is poured onto the back of one of the palms of a panelist. Then these particles are squeezed gently, using forward and backward motions, against the back of the palm with the middle finger and/or the index finger of the other hand. Once the particles are completely spread over the back of the palm, the panelist will rate them for ease of spreadability using the following scale: Unacceptable, Slightly Acceptable and Highly Acceptable.

Cone and Plate Viscosity Measurement

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Scope:

This method covers the measurement of the viscosity of the isotropic phase composition.

Apparatus:

Brookfield Cone and Plate DV-II+ Viscometer;;

Spindle S41;

Plastic cups diameter greater than 2.5 inches.

Procedure:

1. Turn on Water Bath attached to the sample cup of the viscometer. Make sure that it is set for 25°C. Allow temperature readout to stabilize at 25°C before proceeding.
2. With the power to the viscometer off, remove the spindle (S41) by turning counterclockwise.
3. Turn the power on and press any key as requested to autozero the viscometer.
4. When the autozero function is complete, replace the spindle (turning clockwise) and press any key.
5. Attach the sample cup. Using the up/down arrow keys, slowly change the speed to 10 rpm and press the SET SPEED key. Use the SELECT DISPLAY key so that the display is in % mode.

6. Turn the motor on. If the display jumps to 0.4% or higher or will not settle to  $0 \pm 0.1\%$ , turn the adjustment ring clockwise until it does.
- 5 7. Rotate the adjustment ring counterclockwise until the reading is fluctuating between 0.0 and 1.0%. The fluctuation must occur approximately every 6 seconds.
- 10 8. Turn the adjustment ring clockwise exactly the width of one division from the setting reached in step 7.
- 15 9. Turn the motor off. Using the up/down arrow keys, slowly change the speed to 0.5 rpm and press the SET SPEED key. Use the SELECT DISPLAY so that the display is in cP.
- 20 10. Place  $2 \pm 0.1\text{g}$  of product to be measured into the sample cup. Attach the cup to the viscometer.
- 25 11. Allow the product to remain in the cup with the motor OFF for 2 minutes.
- 30 12. Turn the motor ON and allow the spindle to turn for 2 minutes before noting the reading on the display.

While this invention has been described with respect to particular embodiments thereof, it is apparent that numerous other forms and modifications of the invention will be obvious to those skilled in the art. The appended claims and this invention generally should be construed to cover all such obvious forms and modifications which are within the true spirit and scope of the present invention.